

## Procurement of FTIR system for *in situ* studies of epitaxial film growth by atomic layer deposition

### Background

The Power Electronics Branch of the Electronics Science and Technology Division plans to conduct extensive research into low temperature epitaxial growth of thin film materials, III-nitrides, metals and metal oxides, grown by atomic layer epitaxy (ALE). Such materials are critical for future electronic and optoelectronic device technologies needed by the Navy. Progress in said research can be greatly accelerated through employment of *in situ* process characterization methods, in particular characterization of surface chemical reactions at the growth surface are desired. Fourier Transform Infrared Spectroscopy (FTIR) is a proven method for gaining insight into surface chemical reaction mechanisms. This procurement is to obtain an FTIR system that can be effectively integrated with an existing atomic layer epitaxy system in Code 6880 and permit the desired studies. The procured will be integrated with a system currently housed in a laboratory in Building 208 at the Naval Research Laboratory Washington, DC campus.

### General Specifications

The desired FTIR system must be easily and effectively integrated to an existing atomic layer epitaxy reactor that provides two optical access ports located on opposite sides of the reactor chamber and at a seventy (70) degree angles from the surface on which the chemical reactions of interest will take place. It is understood that this puts some unusual requirements on the system. Some typical modes of FTIR are eliminated, including the commonly used transmission mode, since the geometry of installation and the expected signal of sub-monolayer reactant coverage on surfaces of films would be too low to give any information. Instead, a reflection approach must be used to collect information on the surface chemical reactions. The infrared reflection absorption spectroscopy (IRRAS) technique will be necessary for collecting signal from surface species on the relevant substrates, which include sapphire and other semiconductors. As mentioned, the reactor does have two ports at 70 degrees off normal that could be sealed with salt windows that are transparent in the IR region to permit an IRRAS technique to be employed. In this manner and in order to accommodate other physical constraints outside the reactor, the IR source will need to be coupled with fiber optic cable to optics mounted near the window and focused on the sample. The reflected beam, which contains the desired information on surface chemical reaction mechanisms will be collected by a fiber optic bundle with appropriate collection optics and directed into the detector. Because the signal-to-noise ratio is expected to be challenging for this configuration, and for some of the materials of interest, the system should be modular in design and allow for implementation of a probe tip that allows multiple internal reflections (FTIR-ATR) to increase the signal if necessary. The process to be studied can involve pressures in the milliTorr to Torr range of hydrogen, nitrogen, argon and ammonia gases (neutral and plasma activated) and temperatures from room temperature to 500°C. All materials associated with the FTIR system that are inside the reactor must be compatible with such environments.

### Detailed FTIR Specifications

The *in situ* fiber coupled IRRAS system must include:

1. An interferometer module containing:

7. Detailed instruction manual (in English) and free technical support for 2 years.
8. One-year warranty consistent with normal offering to all customers.

To be offered as an options:

1. High temperature ATR Head
  - a. Easily integrated into system specified above to convert a standard probe for use at high temperature.
  - b. Includes integrated water and gas cooling and exposed ZnS ATR crystal with conical tip.
  - c. Integrated type K thermocouple (at probe head).
2. Mid-IR Fiber-Optic Immersion Probe Cable
  - a. 1.2 meter bifurcated probe cable with:
    - i. A 19 fiber bundle of 0.6 NA, 500  $\mu\text{m}$  core/ 600  $\mu\text{m}$  clad chalcogenide fibers (7 input, 12 output).
    - ii. 5000  $\text{cm}^{-1}$  to 870  $\text{cm}^{-1}$  spectral range
    - iii. Designed with internal damping to increase shock and vibration resistance
    - iv. Appropriate bundle connectors on each leg.
3. Advanced Detector module, includes:
  - a. A narrow-band (650-4000  $\text{cm}^{-1}$ ) liquid nitrogen cooled MCT-A detector
  - b. A ZnSe hemispherical immersion lens to optimized fiber coupling
  - c. A 20 hour  $\text{LN}_2$  dewar
  - d. Appropriate lock-down xyz adjustments compatible with system bundle connectors.